Does Thorium Exist as Thorium Silicate in Monazite?

BY

OTTO KRESS, B.S., A.M.

DISSERTATION

SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN THE FACULTY OF PURE SCIENCE OF COLUMBIA UNIVERSITY

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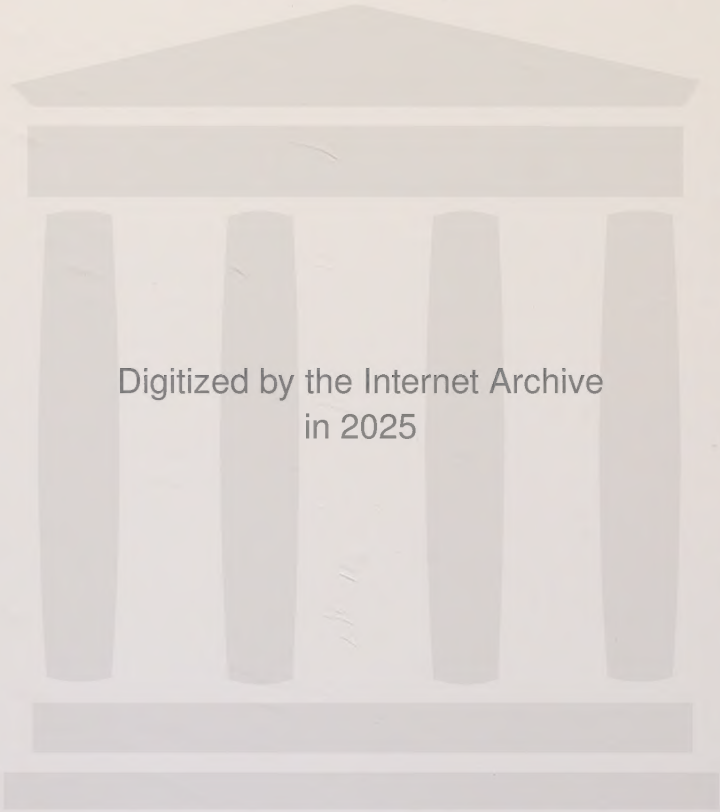
ACKNOWLEDGMENT.

This investigation was carried out under the direction of Dr. Floyd J. Metzger, and I wish here to express my most sincere thanks to him for his kind interest and encouragement in this work, and for the valuable assistance rendered.

O. K.

HAVEMEYER HALL, COLUMBIA UNIVERSITY,
March, 1909.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Does Thorium Exist as Thorium Silicate in Monazite?

MONAZITE, as is well known,is es sentially a phosphate of cerium, lanthanum, neo- and praseo-didymium, with varying amounts of thorium. The form in which the thorium exists in monazite has never been definitely decided, some authorities stating that the thorium is present as a silicate, either as organite or thorite, while others claim that it is present as a phosphate, either replacing a part of the cerium earths or being present as a normal phosphate; still others claiming that it is present both as phosphate and silicate.

The conclusions of the various investigators, while not only differing in view on the nature of the combination of thorium in monazite, are based on insufficient data. In most cases but one or two small samples from different localities, more or less impure, were analyzed.

It has therefore appeared advisable to analyze as many samples of pure crystals of monazite from as many different localities as could be obtained, and to determine, if possible, the combination in which the thorium exists in monazite.

Dunnington [1] from the analysis of one sample of monazite from Amelia County, Va., suggests that the thorium may be combined with the silica as a thorium silicate in the form of orangite.

[1] Am. Chem. Journ. **4**, 1882, p. 138.

Penfield,[1] from the analyses of three specimens of monazite respectively from Portland, Conn., Burke County, N. C., and Amelia County, Va., has deduced the thoria and silica ratio as a 1:1 ratio, and asserts further that thorium, being tetravalent, would not be isomorphous with the cerium earths present in the monazite. His conclusions are, therefore, that the thorium is present in the form of thorium silicate, which exists as an impurity in the monazite.

Blomstrand[2] assigns complex formulæ to the monazite, in which part of the thorium is present as silicate and part as phosphate, while from another analysis he combines all the thorium present as phosphate. His conclusions are that silica is never absent, and where it had not been reported in earlier analyses it had been overlooked.

Ling,[3] in speaking of the sources of thorium, describes it as existing in monazite in the form of a phosphate, as an accessory constituent of monazite.

Dana,[4] in describing monazite writes: "Most analyses show the presence of ThO_2 and SiO_2 usually, but not always in the proper amount to form thorium silicate; that this is mechanically present is not certain, but possible.

Reitinger[5] investigated monazite from two localities in Brazil, one sample being from the diamond sands of Bahia, containing crystals of pure monazite 2–4 mm. long. The second sample, from Bandeirinha, in Minas Geraes in Brazil, from which two samples were picked by hand under the microscope and analyzed. His analyses of the first sample were all made on portions of less than 1 gram in weight. For the second sample of manazite portions of .41 and .46 grams were used. From his analyses he concludes that monazite is an orthophosphate of cerium, lanthanum and didymium, in which a portion of trivalent earth metal has

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[1] Am. Journ. of Science, 3, **24**, 1882, p. 250–54.
[2] Journ. f. Prak. Chemie, **41**, 1890, p. 266–77.
[3] Chem. Zeit. 2, Vol. 19, 1895, p. 1468.
[4] A System of Mineralogy, 6th ed., 1899.
[5] Dissertation, Royal Technical School, Münich, 1902.

been replaced by thorium, and that the theory of Penfield that the thorium belongs to admixed thorite, or that part of the thorium is united with the silica, is incorrect. The empirical formula he assigns to monazite is as follows:

$$X\cdot(Ce, La, Nd, Pr)\cdot yTh_3(PO_4)_4.$$[1]

He states further that the theory that thorium plays the part of a weak acid, replacing zirconium, titanium, silicon, etc., fails because no salts of thorium are formed with the strong alkalis even on fusion.

Böhm[2] describes the thorium as being no definite part of the monazite, but only a fairly regular addition to it, its quantity varying within wide limits.

Experimental Part

PART I

Determination of Thoria and Silica.—The method used for the determination of the thoria was the fumaric acid method originated by Metzger.[3] As slight variations were introduced, especially on account of the silica determination, the method will be described in some detail.

From 1–2.5 grams (seldom less than 1 gram) of the finely ground monazite was heated with concentrated sulphuric acid in a large platinum crucible, with the heat directed downward upon the cover of the crucible for at least ten hours.[4] The contents of the crucible after cooling was poured slowly and with constant stirring, into about 700 cc. of water which had been well cooled in an ice bath. This was allowed to stand over night, filtered, and washed. The residue contained the total silica. If the total silica was

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[1] It is the author's opinion that this formula is a misprint in the original article and that it should have been written

$$X(Ce, La, Nd, Pr)PO_4\cdot yTh_3(PO_4)_4.$$

[2] Seltene Erden, Vol. 2, pp. 89–90.)

[3] Journ. Amer. Chem. Soc., **24**, 901, 1902.)

[4] This method of applying the heat is found to be the most satisfactory, as there is no possible loss due to spattering or creeping of the liquid.

to be determined, this was ignited, weighed, and checked by hydrofluoric and sulphuric acids in the usual manner.

When the quartz and amorphous silica which had been derived from the decomposition of the silicate was to be determined, the method of Lunge and Millberg [1] was used. The filter papers with the total silica were transferred to a platinum dish and digested for fifteen minutes on a water bath with a five per cent solution of sodium carbonate. The quartz being insoluble, was then filtered, ignited, treated with sulphuric acid, reignited, weighed and checked with hydrofluoric and sulphuric acid. The sodium carbonate solution of the silicate silica was acidified with hydrochloric acid, evaporated to dryness, and dehydrated twice, and the silica determined in the usual manner.

The filtrate from the silica determination was then nearly neutralized with ammonia, heated to near the boiling point, and precipitated by an excess of oxalic acid with constant stirring. The precipitate was allowed to stand over night, then filtered and washed with a dilute solution of oxalic acid to prevent the precipitate from running through the paper. The precipitated oxalates were then converted to hydroxides by boiling with strong caustic soda solution, filtered, washed, and dissolved off the filter by means of nitric acid. The solution was then evaporated nearly to dryness on the hot plate, then heated on a water bath with occasional additions of small quantities of water, till all the free nitric acid was expelled.

The residue was then taken up with about 50 cc. of water, 150 cc. of 95 per cent alcohol added, and 60–70 cc. of a saturated solution of fumaric acid was added and the solution heated to boiling on a water bath.

The thorium fumarate was filtered hot, either by suction or in a long-stemmed funnel, washed with hot 40 per cent alcohol and the precipitate and paper returned to the beaker in which the precipitation was made. The thorium

[1] Zeit. f. angew. Chemie, pp. 393–425, 1897.

fumarate was dissolved by heating with 25–35 cc. of dilute hydrochloric acid, filtered to separate the filter paper, and evaporated to dryness and all free acid driven off on a water bath as before. The carbonaceous residue was stirred loose with a rubber-capped rod, taken up with 50 cc. of water, 150 cc. of 95 per cent. alcohol, and precipitated with fumaric acid as usual. The thorium fumarate was then filtered, washed with hot 40 per cent of alcohol, ashed in a weighed platinum crucible, and finally ignited for fifteen minutes with a strong blast and weighed as ThO_2.

The error in the determination of quartz and silicate silica was found by Lunge and Millberg [1] to be from 0.1 to 0.2 per cent of the total silica, by which amount the quartz will appear too low, the amorphous silica too high.

In the analyses of monazite shown in the table below, a number of determinations were made in which total silica checked the sum of quartz and the silicate or amorphous silica within the allowable experimental error. All the silica determinations were, of course, made in platinum vessels.

Samples of monazite crystals from as widely separated localities as could be obtained were analyzed, using the above methods as described, for thorium, total silica, quartz, and amorphous silica, which represents the silica which had been combined in the monazite in the form of silicates. The monazite crystals obtained for analysis varied in weight from .8 to 10 grams, and had all external silicates or minerals other than monazite removed as far as possible by polishing with an emery wheel. Even with these precautions, on breaking the samples preparatory to grinding it could be often seen that the monazite was impure and other minerals were present to a slight extent. A petrographic investigation showed that these impurities when examined were not a thorium silicate such as thorite, but other silicates, oxide of iron, quartz, etc., as will be pointed out later. Wherever the monazite crystals were of sufficient size, that is, weigh-

[1] Loc. cit.

ing at least two grams, the crystal was broken up and two or more separate analyses made, the number depending on the original weight of the crystal. Several analyses made on different fragments of one large crystal, give data which is almost as valuable as though analyses were made on separate crystals, as the composition of different parts of the same monazite crystal varies.

Results of Analyses.

Sample Number.	Weight of Material Taken in Grams.	Percentage Thoria.	Percentage Total Silica.	Percentage Quartz.	Percentage Silicate Silica.	Percentage SiO_2 Required to Form $ThSiO_4$ with ThO_2 Found.	Percentage ThO_2 Required to Form $ThSiO_4$ with Total SiO_2	Percentage ThO_2 Required to Form $ThSiO_4$ with Silicate SiO_2.
1*A*	2.0000	6.45	1.89	.....	.....	1.47	7.49	.....
1*A*	2.0000	6.62	1.84	.....	.....	1.51	8.07	.....
1*A*	2.2252	6.65	1.95	1.3	.65	1.52	.....	2.85
2*A*	2.0000	6.54	1.27	.....	.....	1.49	5.57	.....
2*A*	2.0000	6.60	1.31	.....	.....	1.51	5.73	.....
2*A*	2.0000	6.52	1.28	.....	.....	1.49	5.61	.....
2*A*	2.0000	6.64	1.32	.....	.....	1.51	5.79	.....
2*A*	2.0000	6.57[1]	1.29	.67	.63	1.50	.....	2.74
2*A*	2.0000	6.57[1]	1.29	.69	.60	1.50	.....	2.63
3*A*	2.0000	7.01	1.39	.....	.....	1.60	6.10	.....
3*A*	2.0000	7.31	1.41	.....	.....	1.67	6.40	.....
3*A*	2.0000	7.19	1.46	.....	.....	1.64	6.40	.....
3*A*	2.0000	7.14	1.44	.....	.....	1.63	6.31	.....
3*A*	2.0000	7.16[1]	1.43	.885	.58	1.63	.....	2.54
3*A*	2.0000	7.16[1]	1.43	.875	.52	1.63	.....	2.28
4*A*	2.0000	6.94	1.37	.....	.....	1.58	5.99	.....
4*A*	2.0000	7.08	1.31	.....	.....	1.61	5.72	.....
4*A*	2.0000	7.00	1.37	.....	.....	1.60	5.99	.....
4*A*	2.0000	7.01[1]	.....	.....	.51	1.60	.....	2.24
4*A*	2.0000	7.01[1]	.....	.....	.47	1.60	.....	2.06
5	.9946	9.63	1.04	.....	.....	2.19	4.53	.....
6	.6876	9.14	2.65	.....	.....	2.08	11.62	.....
7*A*	1.4618	12.83	2.65	.....	.....	2.93	11.62	.....
7*B*	2.6070	12.97	2.68	.....	.....	2.96	11.67	.....
7*C*	1.4410	12.81	2.68	.....	.....	2.91	11.67	.....
8	1.4291	8.47	1.79	.....	.....	1.93	7.85	.....

[1] ThO_2 not determined, this being the average of the three determinatives on this sample.

Results of Analyses—*Continued.*

Sample Number.	Weight of Material Taken in Grams.	Percentage Thoria.	Percentage Total Silica.	Percentage Quartz	Percentage Silicate Silica.	Percentage SiO_2 Required to Form $ThSiO_4$ with ThO_2 Found.	Percentage ThO_2 Required to Form $ThSiO_4$ with Total SiO_2.	Percentage ThO_2 Required to Form $ThSiO_4$ with Silicate Silica.
9*A*	1.2780	9.49	1.47	.....	.....	2.16	6.45	.....
9*B*	1.1724	8.26	1.58	.....	.....	1.88	6.85	.....
10*A*	.9934	9.41	1.81	.....	.....	2.14	7.94	.....
10*B*	.8120	11.01	2.46	2.05	1.41	2.51	.....	6.18
11	.8631	8.26	1.03	.08	.95	1.88	.....	4.17
12	.7203	10.13	1.78	.....	.....	2.31	7.80	.....
13	1.3267	11.35	1.57	.....	.....	2.58	6.88	.....
14*A*	1.1721	10.49	3.11	.....	.....	2.39	13.64	.....
14*B*	1.1747	9.99	4.24	1.39	2.85	2.28	.....	12.50
15	1.4381	8.69	3.57	.....	.....	1.98	15.65	.....
16*A*	1.3002	10.81	2.65	.....	.....	2.47	11.62	.....
16*B*	1.3362	11.46	3.07	.51	2.56	2.61	.....	11.23
17*A*	2.3416	8.73	1.66	.03	1.63	1.99	.....	7.15
17*B*	1.7102	7.77	2.76	1.05	1.71	1.77	.....	7.50
18*A*	1.1350	10.73	2.75	.26	2.49	2.45	.....	10.91
18*B*	1.7916	11.27	3.11	.40	2.71	2.57	.....	11.88
18*C*	1.8729	12.72	.....	.....	2.64	2.90	.....	11.57
19*A*	1.4178	16.52	3.70	.24	3.46	3.77	.....	15.17
19*B*	1.3044	15.81	.....	.....	2.68	3.61	.....	11.75
19*C*	1.3374	15.78	3.05	.39	2.66	3.60	.....	11.66
19*D*	1.5102	13.38	3.00	.46	2.54	3.05	.....	11.14
19*E*	1.1018	16.16	3.68	.20	3.48	3.69	.....	15.26
20	1.3985	12.65	3.01	.23	2.78	2.88	.....	12.19
21	1.8260	12.74	2.95	.20	2.75	2.91	.....	12.06
22	1.2684	6.64	2.46	1.00	1.46	1.51	.....	6.40
23	1.7031	12.41	2.82	.71	2.11	2.83	.....	9.25
24*A*	1.7740	9.44	2.72	1.39	1.33	2.15	.....	5.83
24*B*	1.5781	9.40	1.47	.02	1.45	2.14	.....	6.36
25	1.7123	7.87	1.37	.33	1.04	1.79	.....	4.56
26	2.3968	9.34	1.80	.00	1.80	2.13	.....	7.89
27	1.5913	10.12	2.45	.48	1.97	2.31	.....	8.64
28	2.4595	9.36	2.09	.31	1.78	2.13	.....	7.81
29	1.9530	9.63	.....	.....	1.52	2.20	.....	6.67
30*A*	1.9142	7.61	1.84	.12	1.72	1.74	.....	7.54
30*B*	2.0398	8.30	2.06	.53	1.53	1.89	.....	6.71
30*C*	2.0570	7.90	2.26	.51	1.75	1.80	.....	7.67
30*D*	1.6806	7.69	2.02	.18	1.84	1.75	.....	8.07
30*E*	1.7347	7.53	.....	.....	1.95	1.72	.....	8.55

DESCRIPTION OF THE SAMPLES.

Samples 1*A*.[1] Were obtained from the concentrates of North Carolina monazite, the crystals being from $\frac{1}{8}$ to $\frac{1}{4}$ inch long, and could be readily picked by hand from the other associated minerals. On these picked crystals ground to 120 mesh, duplicate determinations were made.

Samples 2*A*. Specimen of North Carolina monazite picked by hand, ground to 120 mesh, and analyzed in duplicate.

Samples 3*A*. Specimen of North Carolina monazite picked by hand, ground to 120 mesh, and analyzed in duplicate.

Samples 4*A*. Specimen of North Carolina monazite picked by hand, ground to 120 mesh, and analyzed in duplicate.

Samples 5 *and* 6. Two crystals imbedded in orthoclase, from Moss, Norway. Crystal 6 was darker than Crystal 5, although imbedded in the same matrix.

Samples 7*A*, 7*B*, 7*C*. Different portions of one large crystal from Moss, Norway. On breaking the mineral, small dark spots were noticed in the fracture. The mineral was therefore not entirely homogeneous.

Sample 8. Single crystal from Moss, Norway, imbedded in the same matrix as Crystal 9*A* and 9*B*, from which separate samples were obtained.

Sample 10*A*, 10*B*. Crystal of monazite, probably from Moss, Norway. The crystal showed on breaking a fracture which was darker in spots than the remainder of the mineral, and was therefore not homogeneous.

Sample 11. Small crystal from Moss, Norway.

Sample 12. Small crystal, locality unknown, probably Moss, Norway.

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[1] The author is indebted to Messrs. M. C. Whitaker, and H. S. Miner, of the Welsbach Light Company, for all samples of North Carolina monazite.

Sample 13. Small crystal, locality unknown, probably Moss, Norway.

Samples 14*A*, 14*B*. One crystal from Arendal, Norway, from which two separate portions were obtained. The The crystal was black in color, and on crushing, preparatory to grinding, showed red streaks in the interior. The red streaks were undoubtedly oxide of iron.

Sample 15. One black crystal from Arendal, Norway. Interior showed red streaks.

Samples 16*A*, 16*B*. Black crystal from Arendal, Norway. Interior showed red streaks, more pronounced than in samples 14*A* and 14*B*.

Samples 17*A*, 17*B*. One homogeneous crystal from Arendal, Norway, on which two determinations were made on separate fragments.

Samples 18*A*, 18*B*, 18*C*. One large crystal from Arendal, Norway. The crystal had altered on the surface and was not quite uniform throughout.

Samples 19*A*, 19*B*, 19*C*, 19*D*, *and* 19*E*. One large crystal from Arendal, Norway.

Sample 20. One black crystal from Arendal, Norway, had altered to brownish color on surface.

Sample 21. One black crystal from Arendal, Norway. Had altered on surface to brownish color. Interior not homogeneous.

Sample 22. One crystal from Arendal, Norway.

Sample 23. One crystal from Arendal, Norway.

Samples 24*A*, 24*B*. One crystal from Arendal, Norway.

Sample 25. One crystal from Arendal, Norway.

Sample 26. One crystal from Arendal, Norway.

Sample 27. The crystal came from Arendal, Norway, and was not homogeneous, containing many dark spots.

Sample 28. One crystal from Arendal, Norway.

Sample 29. One crystal from Arendal, Norway.

Samples 30*A*, 30*B*, 30*C*, 30*D*, *and* 30*E*. One large crystal from Arendal, Norway. The crystal was reddish in

color, with several slightly darker portions. On cracking the crystal appeared homogeneous.

CONCLUSIONS.

From a careful consideration of the analyses of monazite the following conclusions may be derived.

I. In no case was silica absent, although thirty distinct crystals from varying localities had been investigated. Inasmuch as some of these crystals were broken into several distinct fragments, each of which was separately analyzed, the work above can justly be considered as representing the analyses of forty-eight distinct samples.

II. The percentage of silica, while in general it increases with an increase in thoria content, does not do so invariably.

III. Out of a total number of fifty analyses, in which the *silicate silica* was separately determined, forty-five samples show an insufficient silica to combine with the thorium found to form thorium silicate.

IV. Thirty-nine analyses show insufficient *total* silica to combine with the thorium found.

V. Unfortunately, total silica only was determined in samples 6, 14*A*, 15, and 16*A*, which samples show more total silica than is required to combine with the thoria found. In the absence of a silicate silica determination, these results cannot be discussed.

VI. Samples 14*B*, 18*A*, 18*B*, 30*D*, 30*E*, show that the thoria found was insufficient to unite with all the silicate silica. There are here two possibilities: First, that the thorium exists as silicate; second, that the thorium exists as phosphate, and that the silicate silica found came from silicates other than thorium silicate, existing in the monazite as an impurity. It is the opinion of the author that the latter assumption is the correct one, and this is borne out by the microscopic examination of monazite crystals, which constitutes Part II of this paper.

The vast majority of the analyses show insufficient silicate silica to form thorium silicate, and in view of the

fact that thorates have never been prepared, the author believes that the thorium is present as thorium phosphate, thereby agreeing with the views of Reitinger. The silicate silica is probably derived from the decomposition of silicates other than thorium silicate. These silicates were observed in the case of the North Carolina monazite, under the microscope, and were not thorite.

With regard to the complex formulas assigned to monazite by Blomstrand, in which part of the thorium is present as phosphate, and part as silicate, it may be said that the methods available at the present time for the quantitative separation of these rare earths are not sufficiently accurate to warrant the conclusions reached by him.

To sum up briefly, out of the sixty-four analyses made, fifty-five indicate that the thorium is not present as a silicate. Five determinations show an excess of silicate silica over that required by the thorium, and four determinations cannot be considered in the absence of separate silicate silica determinations.

PART II

Microscopic Examination

For assistance in the microscopic investigation of samples of monazite, the author is indebted to Dr. C. P. Berkey, of the Department of Geology of Columbia University. Without his assistance this part of the work could not have been undertaken.

A number of crystals were selected from samples 3*A* and 4*A* (described in Part I), taking only such crystals as appeared to the naked eye to have been unaltered and uniform.

Sections were made of these crystals and examined under the polarizing microscope.

The presence of quartz, a complex silicate, and oxides of iron, were observed in the ground mass of the monazite, as shown in Fig. I.

The large hexagonal crystal observed in Fig. I is quartz, which shows the complex silicate above and to the right of it, the whole being surrounded by monazite.

Under polarized light the quartz and complex silicate are shown in Fig. II. The oxides of iron which filled the crevices and cracks of all the sections could be removed by warming with dilute hydrochloric acid. This is shown in Fig. III, a section from another sample of monazite where the oxides of iron were dissolved away by warming with dilute hydrochloric acid.

In Fig. IV we have the same section after treatment with hydrochloric acid but taken under polarized light.

Figs. III and IV show a section after the oxides of iron had been removed by treatment with acid, Fig. IV being under polarized light. In a few places the section shows holes produced by the fine grinding and the action of the hydrochloric acid.

A few dark spots were observed which do not appear in the figures. These black to brown particles are rounded grains, which appear to be metallic and are wholly enclosed in the mass of the monazite, so that the acid could not act on them. These grains are present to a very small extent in the samples examined, probably less than one per cent, and are due to oxide of iron.

Under all circumstances, nothing was found in any slide which approximated the description of a monazite from Portland, Conn., as given by Penfield[1] as follows: "It showed small grains of a darker resinous substance scattered through the section, which are undoubtedly the mechanically mixed thorite." This description does not fit any of the samples of the monazite examined, but is more descriptive of the thorite as shown in Fig. IV.

Fig. V is another view of a section of monazite, showing the same general characteristics as the other sections. The black bands are due to iron oxides, the cleavage is the same as in other specimens.

[1] Amer. Journ. Science, 3, Vol. 24, 1882, p. 253.

The only mineral other than quartz and oxides of iron, as shown in the sections, is a silicate which is shown in Fig. I and Fig. II.

To determine the nature of this silicate, and to show that it was not thorium silicate, a section was made of thorite for comparison. This section of thorite is shown in Fig. VI. The totally different character of this mineral from any of the observed sections of monazite, is seen at once on comparing Fig. VI with any of the other plates.

On comparing the optical characteristics of the section of the thorite with that of the silicate in the monazite, the following facts were observed:

The thorite is tetragonal and therefore unaxial, and is optically positive.

The silicate observed in the monazite sections showed distinct cleavage, probably two, one of which was more pronounced than the other. It is biaxial and optically negative. It shows a very low extinction angle.

The index of refraction is a little higher than that of quartz and a little lower than that of monazite, while the double refraction is low.

These optical properties at once show that the silicate observed in the monazite is not a thorium silicate or thorite, but is probably feldspar.

The microscopic investigation therefore bears out the chemical investigation that thorium is not present in monazite in the form of thorium silicate.

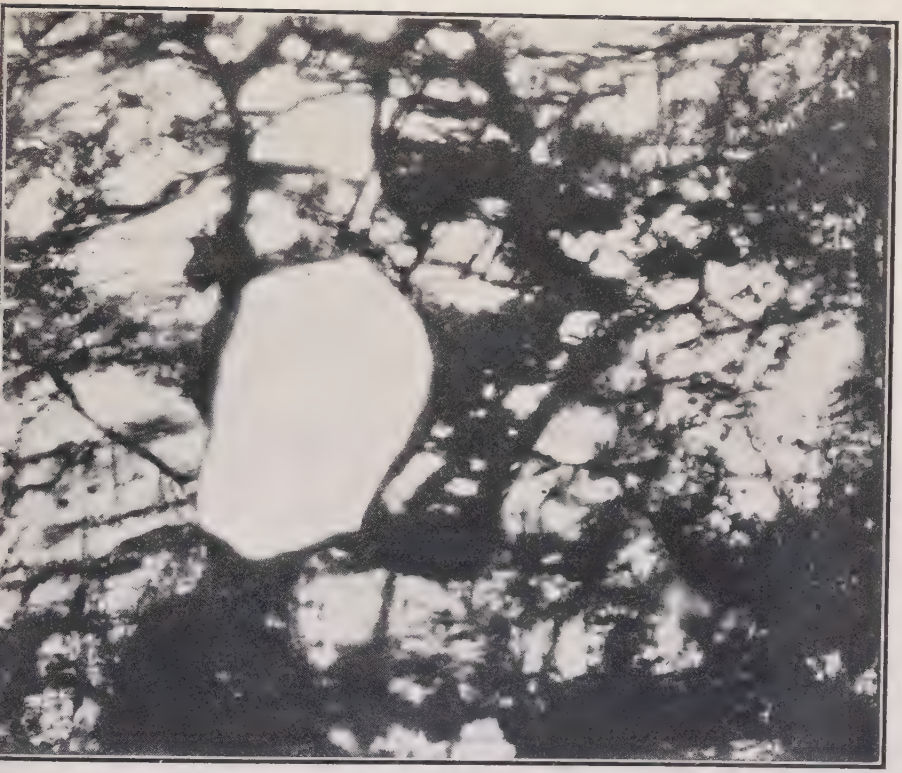

FIG. I.—Plate shows a large crystal of quartz, the silicate immediately above and to the right of it and the monazite. ×70.

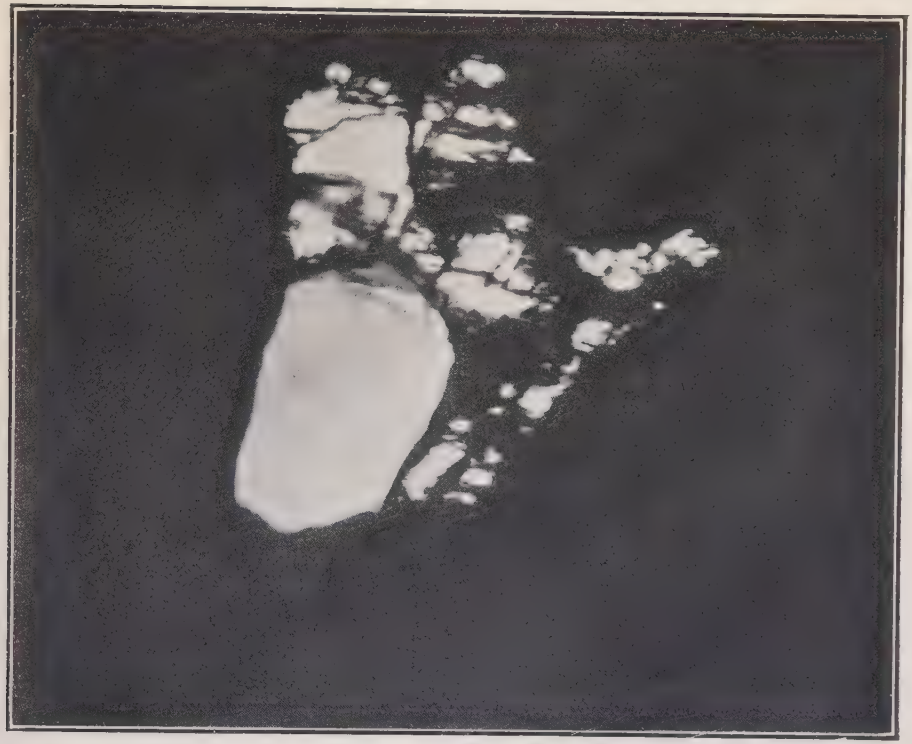

FIG. II.—The same as Fig. I, but under polarized light. Plate shows the crystal of quartz, the silicate, and the monazite which appears black here. ×70.

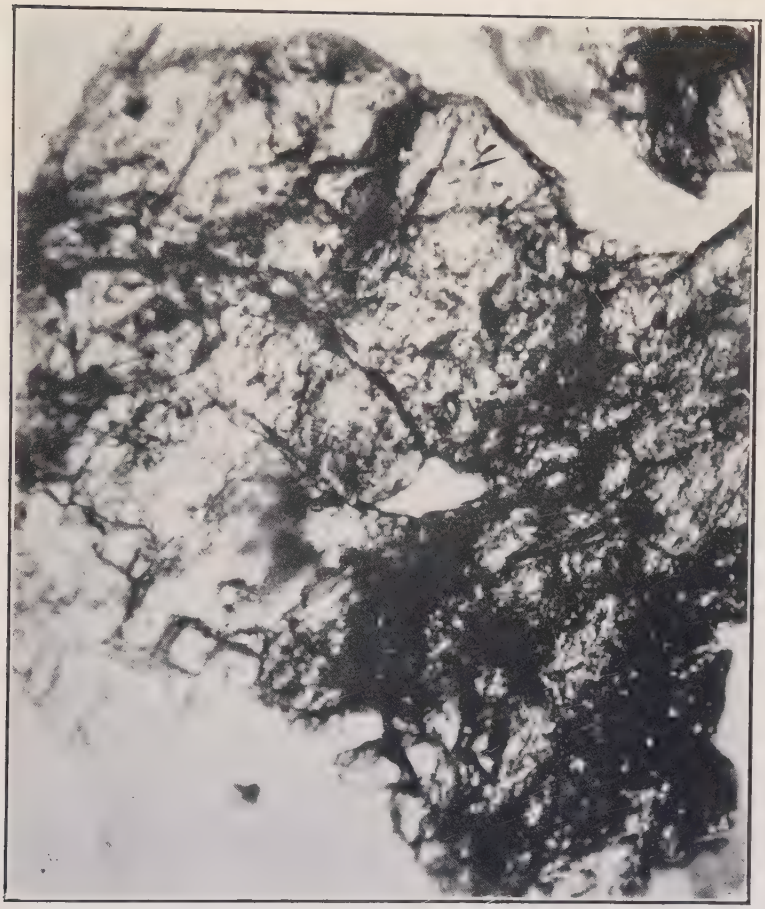

FIG. III.—Plate showing a section of monazite after treatment with hydrochloric acid. ×70.

FIG. IV.—Same as section Fig. III, shown under polarized light. ×70.

Fig. V.—Plate showing another section of monazite. ×70.

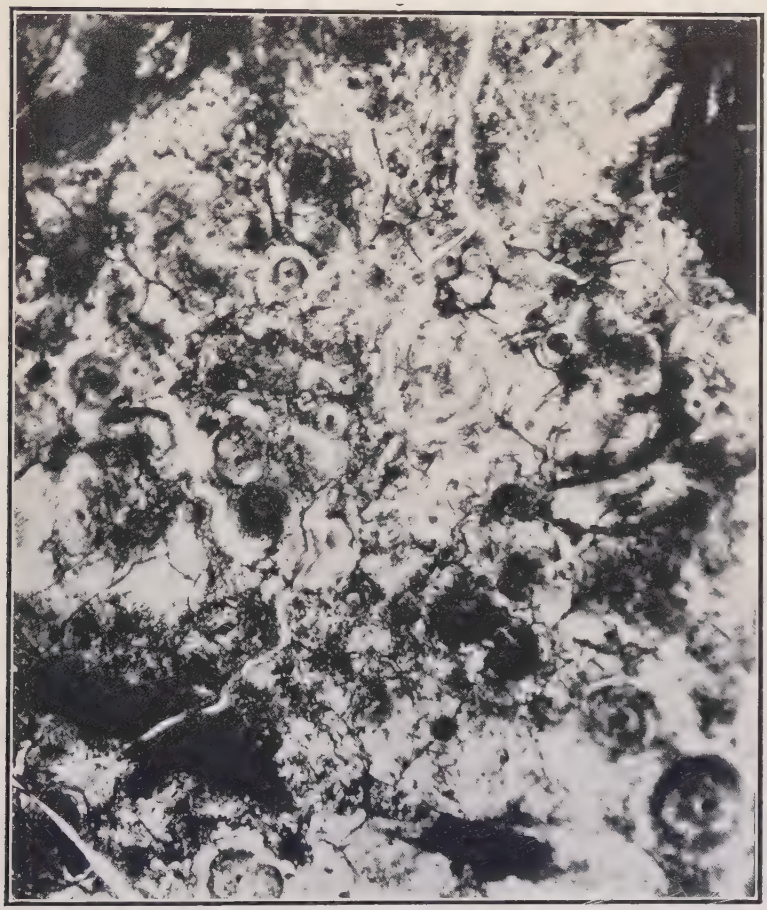

FIG. VI.—Section of thorite. ×70.

BIOGRAPHICAL.

Otto Kress entered Columbia University in September, 1902, and was graduated June, 1906, receiving the degree of B.S.

From 1906–1907 studied under the Faculty of Pure Science at Columbia University, and received the degree of A.M.

From September, 1908, to June, 1909, studied under the Faculty of Pure Science for the degree of Ph.D.